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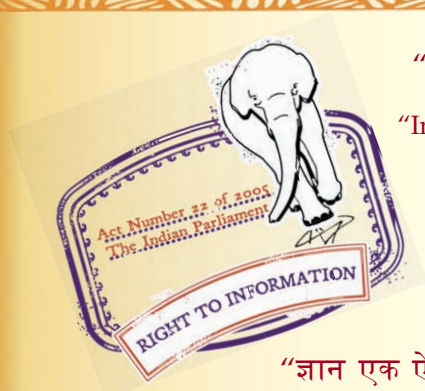
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IS 6155 (1987): Sodium carbonate, monohydrate, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 6155 - 1987

Indian Standard

SPECIFICATION FOR
SODIUM CARBONATE, MONOHYDRATE,
PHOTOGRAPHIC GRADE
(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SODIUM CARBONATE, MONOHYDRATE, PHOTOGRAPHIC GRADE (First Revision)

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(Continued from page 1)

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Indian Standard
SPECIFICATION FOR
SODIUM CARBONATE, MONOHYDRATE,
PHOTOGRAPHIC GRADE
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 30 June 1987, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This Indian Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality and to furnish the manufacturers, suppliers and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

0.3 Sodium carbonate, monohydrate is used in the processing of sensitized photographic materials. It finds use in preparing photographic developers, particularly in colour films where high purity of the ingredients is very essential and extremely important. It has a chemical formula $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ and molecular mass 124.

0.4 This Indian Standard was first published by the Acids, Alkali and Halides Sectional Committee in 1971 and had covered the material for use in pharmaceutical and antibiotic industry along with that for photographic industry. In the present revision, the material suitable for only photographic industry has been covered and accordingly, the title of the standard has also been modified. In the present standard, the requirement for assay has been modified and the requirements for free alkali, bicarbonate and reaction to ammonical silver nitrate have been added.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in

accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for sodium carbonate, monohydrate, photographic grade used in processing of sensitized photographic materials.

2. REQUIREMENTS

2.1 Description — Sodium carbonate, monohydrate, photographic grade shall be in the form of white crystalline free flowing granules, free from lumps and external visible impurities.

2.2 Solution in Water — 20 g of the material dissolved in 100 ml of distilled water at room temperature shall give a clear and colourless solution free from sediments other than a slight flocculence.

2.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods given in Appendix A.

TABLE 1 REQUIREMENTS FOR SODIUM CARBONATE, MONOHYDRATE, PHOTOGRAPHIC GRADE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REFERENCE TO CLAUSE NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay (as Na_2CO_3), percent by mass, <i>Min</i>	85.0	A-2
ii)	Bicarbonates (as NaHCO_3), percent by mass, <i>Max</i>	0.4	A-3
iii)	Free alkali (as NaOH), percent by mass, <i>Max</i>	0.2	A-4
iv)	Reaction to ammoniacal silver nitrate	To pass the test	A-5
v)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.03	A-6
vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.001	A-7
vii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	A-8

*Rules for rounding off numerical values (revised).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in air-tight containers with a plastic liner or as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed and marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Mass of the material in the package;
- c) Name and trade-mark, if any, of the manufacturer;
- d) Lot or batch number; and
- e) Date of manufacture.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or processors may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

A P P E N D I X A

(Clause 2.3)

METHODS OF TEST FOR SODIUM CARBONATE, MONOHYDRATE, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (second revision).

A-2. DETERMINATION OF ASSAY**A-2.1 Reagents**

A-2.1.1 Standard Hydrochloric Acid — 1 N.

A-2.1.2 Methyl Orange Indicator — Dissolve 0.1 g of methyl orange indicator in 100 ml of water.

A-2.2 Procedure — Weigh accurately about 1 g of the material and dissolve in 50 ml of water. Titrate slowly with 1N hydrochloric acid using methyl orange as an indicator.

A-2.3 Calculation

$$\frac{\text{Total alkalinity (as Na}_2\text{CO}_3 \text{)}}{\text{percent by mass}} = \frac{5.3 V N}{M}$$

where

V = volume in ml of standard hydrochloric acid used in titration,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF BICARBONATES**A-3.1 Reagents**

A-3.1.1 Barium Chloride Solution — 1 N, neutral to phenolphthalein.

A-3.1.2 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 50 ml of rectified spirit conforming to IS : 323-1959*, and dilute to 100 ml.

A-3.1.3 Standard Sodium Hydroxide solution — 0.1 N.

A-3.2 Procedure — Weigh accurately about 2 g of the material into a 100-ml conical flask. Add 30 ml of carbon dioxide free water; stopper the flask and dissolve the material. Add 25 ml of barium chloride solution and 10 drops of phenolphthalein indicator. Stopper and swirl. If the solution is colourless (*see* Note), titrate with standard sodium hydroxide solution till the first appearance of a faint pink colour which persists for 30 seconds.

NOTE — If the solution becomes pink, report bicarbonates as absent.

A-3.2.1 The material shall be taken to have not exceeded the limit prescribed in Table 1 if not more than 1 ml of standard sodium hydroxide solution is required in the test.

*Specification for rectified spirit (*revised*).

A-4. DETERMINATION OF FREE ALKALI

A-4.1 Reagent

A-4.1.1 *Standard Hydrochloric Acid* — 0.1 N.

A-4.2 Procedure — If the solution has a pink colour after the addition of the indicator in A-3.2, titrate with standard hydrochloric acid to a colourless end point.

A-4.3 The material shall be taken to have not exceeded the limit prescribed in Table 1, if not more than 1 ml of standard hydrochloric acid is required in the test.

A-5. TEST FOR REACTION TO AMMONIACAL SILVER NITRATE

A-5.1 Reagents

A-5.1.1 *Concentrated Ammonium Hydroxide* — 20 percent (m/m).

A-5.1.2 *Ammoniacal Silver Nitrate Solution* — Prepared by mixing equal volumes of concentrated ammonium hydroxide and 10 percent silver nitrate solution.

A-5.2 Procedure — Dissolve 1.00 g of the material in 20 ml of water. Add 10 ml of freshly prepared ammoniacal silver nitrate solution, and mix well. Let it stand for 2 minutes and compare with a blank prepared simultaneously by adding 5 ml of concentrated ammonium hydroxide to a solution of 1 g of the material dissolved in 25 ml of water.

A-5.2.1 The material shall be taken to have passed the test if any colour or precipitate produced with the material is not greater than that produced in the blank test.

CAUTION — Dispose of all test solutions and rinse the used apparatus immediately. Explosive compounds may be formed on standing.

A-6. DETERMINATION OF CHLORIDES

A-6.1 Reagents

A-6.1.1 *Dilute Nitric Acid* — approximately 4 N and free from chlorides.

A-6.1.2 *Silver Nitrate Solution* — 10 percent (m/v).

A-6.1.3 *Standard Chloride Solution* — Dissolve 0.1649 g of sodium chloride (NaCl) in water and make up the volume to 100 ml in a volumetric flask. Dilute 10 ml of this solution to 1 000 ml in a volumetric flask. One millilitre of this solution contains 0.01 mg of chloride (as Cl).

A-6.2 Procedure — Dissolve 1 g of accurately weighed sample in 100 ml of water in a volumetric flask. Take 10 ml aliquot into a Nessler cylinder and neutralize with dilute nitric acid and add a little excess. Add 1 ml of silver nitrate solution, dilute to 50 ml with water and mix well. Any turbidity produced shall not be greater than a control containing 3 ml of standard chloride solution treated in the same manner as the sample aliquot.

A-7. DETERMINATION OF IRON

A-7.1 Apparatus

A-7.1.1 Nessler Cylinders — 50-ml capacity.

A-7.2 Reagents

A-7.2.1 Hydrochloric Acid — approximately 15 percent (m/v).

A-7.2.2 Ammonium Persulphate

A-7.2.3 Potassium Thiocyanate Solution — approximately 5 percent.

A-7.2.4 Dilute Sulphuric Acid — approximately 10 percent (v/v).

A-7.2.5 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Dilute 100 ml of the solution again to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-7.3 Procedure — Weigh accurately 1 g of the material and dissolve in about 20 ml of water. Add about 5 ml of hydrochloric acid and evaporate to dryness. Dissolve the residue in 20 ml of water. Add 30 mg of ammonium persulphate, and boil to oxidize the iron. Cool and transfer to a Nessler cylinder; add 5 ml of potassium thiocyanate solution, dilute with water to the 50-ml mark and stir well. Carry out a control test in another Nessler cylinder, using 1 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume as of the reaction mixture. Compare the intensity of the colour produced in the cylinders after 5 minutes.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-8. DETERMINATION OF HEAVY METALS

A-8.1 Apparatus

A-8.1.1 Nessler Cylinders — 50-ml capacity.

A-8.2 Reagents

A-8.2.1 Dilute Hydrochloric Acid — approximately 5 N, and 1 : 99 (v/v).

A-8.2.2 *p*-Nitrophenol Indicator Solution — 0.25 percent.

A-8.2.3 Dilute Ammonium Hydroxide — 1 : 99 (v/v).

A-8.2.4 Hydrogen Sulphide Solution — saturated.

A-8.2.5 Standard Lead Solution — Weigh accurately 1.60 g of lead nitrate and add 50 ml of nitric acid of analytical reagent grade. Dissolve in water and make up the solution to 1 000-ml mark. From this, pipette out 10 ml of the solution and dilute it with water to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-8.3 Procedure — Weigh accurately 1 g of the sample and dissolve in 20 ml of water. Add 5 ml of hydrochloric acid (5 N). Evaporate to dryness. Dissolve the residue in 20 ml of water. Add 1 drop of *p*-nitrophenol indicator solution and then add, dropwise, dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid (1 : 99), dropwise, until the solution becomes colourless and then add 0.5 ml in excess. Transfer to a Nessler cylinder; add 5 ml of hydrogen sulphide solution, dilute to 50-ml mark and shake well. Carry out a control test using 1 ml of standard lead solution in place of the material and the same quantities of the other reagents. Compare the intensity of the colour produced in the two Nessler cylinders.

A-8.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A P P E N D I X B

(Clause 4.1)

SAMPLING OF SODIUM CARBONATE, MONOHYDRATE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are nearly filled by the sample.

B-1.6 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment of the material, all the containers of same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers (n) to be selected for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

B-2.3 These containers shall be selected at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS : 4905-1968* may be followed.

*Methods for random sampling.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument, a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for the characteristics given in Table 1 and should be about 400 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 800 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.6**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as a referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see B-3.1.2*) and a set of individual samples (*see B-3.1.3*) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of assay (as Na_2CO_3), percent by mass, shall be conducted on each of the individual samples.

B-4.2 Tests for all other characteristics given in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the individual test results of assay, the average (\bar{X}) and range (R) shall be calculated, where

$$\text{Average (} \bar{X} \text{)} = \frac{\text{Sum of test results}}{\text{Number of tests}}$$

Range (R) = Difference between the maximum and the minimum values of test results.

The lot shall be considered to have passed the requirements of assay content if the value of the expression ($\bar{X} - 0.6 R$) is greater than or equal to the minimum value specified in Table 1.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the requirements specified in Table 1.

B-5.3 The lot shall be declared as conforming to the specification of this standard if **B-5.1** and **B-5.2** are satisfied.

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